

# **EXHIBIT “26”**

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GASKET-FORMING COMPOSITIONS FOR CONTAINER CLOSURES

Filed Nov. 29, 1966

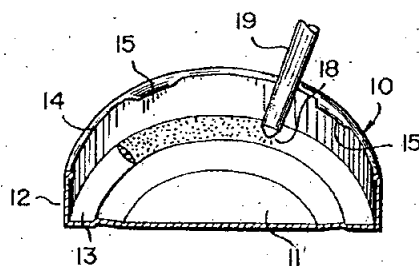


FIG. 1

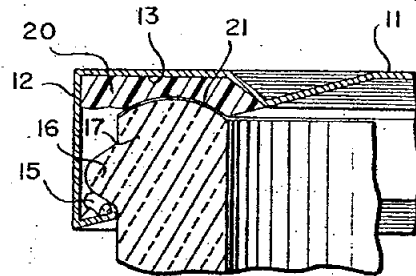


FIG. 2

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1

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## GASKET-FORMING COMPOSITIONS FOR CONTAINER CLOSURES

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3 Claims. (Cl. 215—40)

### ABSTRACT OF THE DISCLOSURE

Compositions for forming sealing gaskets in rotatable container closures which are composed of a vinyl resin, a plasticizer for the resin, and a minor amount of propylene glycol monoricinoleate. The ricinoleate controls the torque which is required to remove the closure from a hermetically-sealed container.

This invention relates to compositions suitable for use as gaskets in rotatable closure elements. In a particular aspect, it relates to sealing compositions containing an additive from which gaskets are formed in closures to provide them with improved removal torque values.

Rotatable closures are usually made of metal, such as lacquered tinplate or aluminum, and have gained extensive use in protecting and preserving foods in glass containers. To be effective, the closure requires a gasket to easily seal the contained food, and it must not include any deleterious substance that might transfer to and contaminate the food. Typical rotatable closures include the screw type and lug type, and they differ chiefly in the means by which the closure is held firmly in place on the container. Illustrative means include a continuous or discontinuous thread, projecting lugs, etc., located near the container opening, and they are adapted to mate in threaded engagement with the closure as it is rotatably advanced to bring the gasket into sealing relationship with the mouth of the container.

In sealing a container with a closure, air is exhausted from the headspace above the contents in closing machines which produces a vacuum either mechanically or by the condensation of steam. In closing the container, the torque must be sufficient to resist retractive movement during shipment and/or storage as such movement is apt to break the seal and cause leaks through which spoilage organisms can gain access to the contents in the container.

The torque which is required for closure removals on vacuum-packed containers varies widely, but it is well known that some containers have their caps so firmly secured that it is necessary to resort to fairly drastic means to effect their removal. On the other hand, a closure may be so loosely attached that only a slight twist is required to remove it from the container. This is objectionable because the container is subject to tampering while stored on shelves by customers who remove the closure to examine the contents and then replace the container on the shelf. It is apparent that such opening breaks the seal and exposes the contents to the atmosphere with the result that spoilage will gradually occur. Another objection is that a loosely-fitted closure may be accidentally jarred, causing the seal to be broken and concomitantly therewith result in exposure of the contents to contamination.

It is, therefore, an object of this invention to provide a composition which is suitable for use as a gasket in a rotatable closure which maintains a hermetic seal but has low removal torque requirements. This objective is achieved by incorporating propylene glycol monoricinoleate as a torque control agent in the gasket-forming composition.

Closure manufacturers design their gaskets with the

2

prime consideration of preventing accidental dislodgement of the closure. This requires formulation of the gasket-forming composition so that it exhibits considerable friction to the closure and the container against rotational torque. But, this is precisely what makes these closures difficult to open for the average consumer. By including propylene glycol mono-ricinoleate in the gasket, the frictional forces which exist between the gasket and the container are appreciably reduced without impairing the sealing efficiency.

Plastisols are frequently used to form gaskets for closures. These compositions basically comprise a vinyl resin dispersed in a plasticizer in which the resin is insoluble at room temperature but which is capable of solvating the resin at an elevated temperature. The properties of these compositions may be modified by the addition of conventional ingredients, such as fillers, stabilizers, pigments and other additives.

The propylene glycol monoricinoleate which is included in the plastisol composition is largely incompatible with the resin and the plasticizer. It is possessed of limited compatibility however, so that it remains essentially within the body of the gasket when it is relaxed or uncompressed. However, a portion of the ester exists as a thin film over the surface of the gasket. As the gasket is subjected to pressure when the closure is brought into sealing relationship with a container, an additional portion of the propylene glycol monoricinoleate migrates to the surface of the gasket and thereby reduces the friction between the gasket and the lip of a container.

In the drawing:

FIG. 1 is a perspective sectional view showing the application of a gasket-forming composition by means of a nozzle in the peripheral channel of an inverted lug-type closure.

FIG. 2 is a fragmentary vertical sectional view of a closure in sealed relationship with a container.

The closure, shown generally at 10, comprises a circular panel 11 and a skirt 12 depending from the periphery of the panel. A gasket-receiving channel 13 is provided in the primetrical margin of the panel adjacent the lower edge of the skirt. The upper edge of the skirt is curved inwardly to form a bead 14 into which are formed a series of spaced lugs 15. These lugs register with the thread 16 (shown in FIG. 2) formed on the neck of the container 17 and lock the closure in place.

The inner surface of the closure is usually coated with a protective film of a varnish or a lacquer, and the gaskets are generally formed from liquid compositions by a lining technique. In the lining operation (FIG. 1), the closure is positioned over a rotating chuck (not shown), and the composition 18 flows through a nozzle 19 into the channel 13. The closure is spun by the chuck, and the composition is distributed as a narrow band in the channel. The lined closures are then passed to a heated oven maintained at a temperature sufficiently high to flux the composition which, when cooled, solidifies to a permanent rubber-like gasket 20 (as shown in FIG. 2). The fluxing step is generally carried out by continuously advancing the lined closures through an oven on a wire mesh belt, and the advance is so synchronized that a residence time in the oven of about 60 second is sufficient to flux the composition.

In applying a gasketed closure to a container, the gasket surface has a thin film of the additive and as the closure is progressively advanced on the container, the increased pressure results in a corresponding increase in the exudation of additive to the surface. The greater the pressure the greater is the exudation, and migration of the additive continues until the closure is brought into sealing contact with the container. The surface layer of additive, shown as an exaggerated film 21 in FIG. 2, remains on the gasket

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3

so long as the sealing relationship is maintained. The additive thus acts as a lubricant by reducing the frictional forces which exist between it and the container and consequently, less torque is required to remove the closure. As the closure is progressively removed, the additive is gradually reabsorbed into the body of the gasket as the gasket is brought out of contact with the container.

Vinyl resin plastisols are particularly suitable for use in forming the gaskets. These include plasticized homopolymers of vinyl chloride and copolymers of vinyl chloride with up to 20% by weight of another monomer copolymerizable therewith. Suitable monomers include acrylonitrile, vinylidene chloride, vinyl acetate, and dialkyl maleates. Typical copolymers include 95 percent vinyl chloride-5% vinyl acetate; 95 percent vinyl chloride-5% dialkyl maleate; and 94 percent vinyl chloride-6% vinylidene chloride. The preferred resin is plasticized polyvinyl chloride.

The plasticizers which may be used in the gasket-forming compositions include dialkyl phthalates, alkyl phthalyl alkyl glycolates, dialkyl esters of alkane dicarboxylic acids, acetyl trialkyl citrates, and trialkyl and triaryl phosphates. Particular plasticizers include dioctyl phthalate (di-2-ethylhexyl phthalate), octyl decyl phthalate, ethyl phthalyl ethyl glycolate, butyl phthalyl butyl glycolate, diisobutyl adipate, dibutyl sebacate, acetyl dibutyl citrate, trioctyl phosphate and tricresyl phosphate. Other useful plasticizers include alkyl esters of fatty acids, such as octyl stearate; epoxidized triglycerides, such as epoxidized soybean oil; and polymeric polyester plasticizers, such as polymeric glycol adipate.

Various other additives may be included to modify the plastisol compositions. These include fillers, such as anhydrous calcium sulfate, talc, wood flour, diatomaceous earth and asbestos; stabilizers, such as tetrasodium pyrophosphate, tribasic lead silicate, calcium stearate, zinc stearate, dibasic lead stearate and organo-tin complexes; pigments, such as carbon black, titanium dioxide and aluminum powder; and dispersing agents such as zinc resinate, lecithin, glycol stearate, propylene glycol laurate and glycerol monooleate.

The propylene glycol monoricinoleate is included in the gasket-forming composition in an amount sufficient to insure a proper balance of lubricating properties in the resulting gasket and will vary with the relative proportions and properties of the other components. In general, the amount should range between about 1 and 18, preferably between about 4 and 12, parts by weight per hundred parts of resin. Unless an excessive quantity is used, the amount that will migrate with time is negligible and the surface of the gasket will remain substantially tack-free.

This invention is further illustrated by the following example.

#### Example

Ingredient:	Parts by wt.
Polyvinyl chloride (dispersion grade) -----	100
Epoxidized soybean oil -----	10.5
Dioctyl phthalate -----	63

4

Barium sulfate -----	100
Titanium dioxide -----	4
Propylene glycol monoricinoleate -----	7

1850 mg. of the above composition was deposited in the annular channel of a rotatable lug-type closure having a diameter of 63 mm. and then fluxed in an oven at a temperature of 400° F. and a residence time of 1 minute. The closure containing the fluxed gasket was placed on a glass jar containing pickles. The pickles were cold filled (75° F.), steam closed, and thereafter processed for 30 minutes in a bath of boiling water. The turn-down torque to seal the container was 25 inch-lbs., which is customarily employed in commercial closure-applying machines, and the container was then stored for three days at a temperature of 100° F.

Following the storage period, the closure was tested for ease of removal, which required a torque of 40 inch-lbs. On the other hand, gaskets formed from identical compositions which did not include propylene glycol monoricinoleate required a removal torque of well over 100 inch-lbs. This is significant in that the frictional forces between the gasketed closure and a container are reduced without adversely affecting the seal.

It had also been observed that the addition of the ester to the composition did not cause corrosion of the metal closure nor cause objectionable staining of the gasket. A further advantage resulting from the addition of the ester to the composition is that adhesion of the gasket to the metal closure was excellent, and no lifting of the gasket resulted when the closure was removed from the container. The amount of propylene glycol monoricinoleate should not exceed 18 parts per hundred parts by weight of resin because quantities in excess of this amount result in a loosely-fit closure.

I claim:

1. A closure comprising a cap adapted to be rotatably attached to a container opening and a rubber-like gasket positioned in the cap to register in sealing relationship with the lip of the container, said gasket comprising a fluxed plastisol of a vinyl chloride resin containing between about 1 and 18 parts by weight of propylene glycol monoricinoleate based on 100 parts by weight of the resin.

2. A closure according to claim 1 wherein the propylene glycol monoricinoleate is present in amounts ranging between 4 and 12 parts by weight.

3. A closure according to claim 1 wherein the polymer is polyvinyl chloride.

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JULIUS FROME, Primary Examiner.

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( 58 of 59 )

**United States Patent****3,971,785****Messina , et al.****July 27, 1976****Process for the manufacture of flowed-in aerosol can gaskets****Abstract**

Process of lining aerosol can valve mounting cups with a gasketing composition comprising a carbon dioxide blocked polyamine dispersed in a liquid polyurethane prepolymer consisting of a free isocyanate group-containing reaction product of a polyisocyanate compound with a polyhydroxy material. The lined cups are heated for a few minutes to gel the gasket and full cure subsequently takes place without further intervention by means of ambient atmospheric moisture. The process is rapid, economical in equipment and produces gaskets which permit a significant extension of swaging specifications.

**Inventors:** Messina; Thomas A. (Waltham, MA); Droney; Patrick J. (Arlington, MA)**Assignee:** W. R. Grace & Co. (Cambridge, MA)**Appl. No.:** 549564**Filed:** February 13, 1975**Current U.S. Class:****264/268; 264/DIG59****Intern'l Class:****B29C 025/00****Field of Search:****264/268,259,DIG. 59,299,345 260/75 NH,77.5 AM,2.5 AM****References Cited [Referenced By]****U.S. Patent Documents**

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<u>3635908</u>	Jan., 1972	Vogt et al.	260/77.
<u>3834578</u>	Sep., 1974	Smith et al.	260/2.

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Primary Examiner: Hoag; Willard E.

Attorney, Agent or Firm: McMillan; Armand

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*Parent Case Text*

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This is a continuation of application Ser. No. 302,005 filed Oct. 30, 1972 and now abandoned.

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*Claims*

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What is claimed is:

1. A method for lining an aerosol can mounting cup with a sealing gasket consisting of:
    - a. depositing in the annular channel of the mounting cup a layer of a composition comprising (1) a liquid prepolymer of a free isocyanate groups-containing reaction product of a polyisocyanate with a polyhydroxy compound and (2) a finely divided carbon dioxide blocked polyamine;
    - b. heating the cup and said composition deposited therein to a temperature sufficient to cause decomposition of said polyamine with time, but said heating being for a time sufficient to gel said composition only and insufficient to cause substantial decomposition of said polyamine; and
    - c. then allowing said composition to cure further at ambient temperature in an atmosphere containing moisture.
  2. The method of claim 1 wherein said heating temperature is under 200.degree.F and said heating time is about 2 minutes.
  3. The method of claim 1 wherein the polyhydroxy compound is a mixture of at least one diol and one triol, said triol constituting at least 30% of the mixture by weight.
  4. The method of claim 1 wherein the polyamine is a triamine and the polyisocyanate is aromatic or alicyclic.
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*Description*

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THE PRIOR ART

Pressurized aerosol containers are widely used for packaging and dispensing an ever larger variety of materials. In this type of application, the product to be dispensed is confined under pressure by means of a volatile propellant and is discharged in a controlled manner through a valve-actuated orifice. A typical aerosol unit comprises a hollow cylindrical container closed at one end and provided with an opening at its opposite end for receiving a dispensing valve assembly. A metal fitment or mounting

CDD12504

cup serves as a closure for the container and also as a support for the valve assembly which is tightly fitted within an aperture centrally located in the cup.

The cup is swaged into the 1-inch standard opening of the aerosol can to produce a container that can safely withstand the internal pressure of the finished package and yet allows the convenient dispensing of the contents when the valve is actuated. An elastomeric sealing material, formed into a gasket by high speed deposition into the annular channel of the mounting cup, compensates for the failure of the metal cup and can to mate perfectly at their juncture. The imperfect metal to metal juncture is the normal result of tolerances, in parts and in swaging process, which must be accepted in the production of an economically feasible aerosol package. Both the manufacturer of cup, valve and can and the aerosol packager who fills and swages the cup into the can must operate within certain allowable tolerances in order that a functional package be produced. In the swaging operation, the parameters of interest are known as crimping dimensions and specified as crimp depth and crimp diameter.

It has been found that the use of a polyurethane composition containing a polyamine carbamate, as the material for the sealing gaskets, has extended the allowable limits of the crimping dimensions beyond those permissible with conventional aerosol mounting cup systems such as the polychloroprene compositions described in U.S. Pat. No. 3,389,113.

The use of polyurethane gaskets per se in aerosol cans was first claimed by Siebel in British patent specification No. 1,203,384 in Oct. 1970. However, the practical problems associated with the high speed placement of gaskets into very large numbers of aerosol mounting cups have effectively kept this type of material from the industrial scene. Part of the reason for this lies in the alternative of using an active curing agent such as a polyamine, or omitting the curing agent and allowing the gasketing material to cure by means of ambient moisture. In the first instance, the inconvenience of a two-package system effectively discourages the cup manufacturer, while in the second instance relatively long cure times of 60 hours or more are needed, causing the accumulation of very large numbers of cups "in process" and thus increasing the requirements for equipment as well as the delay of quality control procedures which must be carried out on the gelled gaskets.

Furthermore, while carbon dioxide-blocked polyamines (carbamates) have been used as curing agents for polyurethane prepolymers to permit the formulation of stable one-package compositions that can be heat activated as desired, the potential advantages of such systems in aerosol mounting cup flowed-in gasket applications have remained largely unexplored. This state of affairs is perhaps due in part to the preponderant selection of diamine carbamates for curing linear polyurethanes destined to the production of filaments, and also in part to the undesirable release of carbon dioxide on heating. In any event, applicants have now discovered unexpected advantages, such as the extension of swaging tolerances already mentioned, in the use of certain polyurethane compositions for aerosol gasketing. These advantages, as well as others, will be fully described in the following disclosure.

## SUMMARY OF THE INVENTION

According to the present invention, it has now become both possible and desirable to line aerosol can mounting cups with exceptional gaskets by a process which involves (1) the high speed placement into the annular channel of the cups of a stable one-package composition comprising a free-isocyanate group-containing reaction product of a polyhydroxy material with a polyisocyanate and a finely divided carbamate which is a carbon dioxide-blocked polyamine, (2) the heating of the flowed-in composition for a few minutes at the decomposition temperature of the carbamate to cause the gelling of the gasket material, (3) the subsequent curing of the gelled gasket material by ambient moisture

CDD12505

during shipment to the aerosol packager, and (4) the swaging of the lined cups to the body of aerosol cans after the latter have been filled with the product to be dispensed.

The carbamate gelled, ambient moisture curing polyurethane aerosol cup gasketing system of the invention provides for, as shall be demonstrated, a substantial widening of allowable tolerances of crimping dimensions beyond those currently permitted in such an assembly, and a means of rapidly bringing a liquid prepolymer resin gasketing compound to a gelled and partially cured state sufficient so that the production quality of resulting gasket-cup assemblies can be observed within 5 minutes of the compound placement.

## DESCRIPTION OF THE DRAWINGS

The present invention will be more clearly understood on reference to the accompanying drawing and the discussion relating thereto:

FIG. 1 is a side elevational view of a typical aerosol container provided with a mounting cup and valve unit.

FIG. 2 is an axial sectional view of the aerosol mounting cup of FIG. 1 carrying a gasket prepared from compositions of the present invention.

FIG. 3 is a fragmentary axial sectional view of the upper portion of the aerosol container of FIG. 1 showing the gasketed mounting cup of FIG. 2 crimped in position over the mouth of the container.

Referring to FIG. 1, a typical aerosol container is illustrated which comprises a cylindrical body portion 10 fitted with a domed top portion 12 and a bottom closure 11. The container is provided with a metering valve generally designated at 13 which is actuated by button 15. The actuator button is carried on a hollow valve stem 14 and provided with an orifice 16 through which the container contents are discharged when valve 13 is opened. Communicating with valve 13 is a dip tube 17 of sufficient length to allow the contents to be discharged from the container. The valve unit, which may be any of the various types known to the art, is supported by a mounting cup, generally designated at 18, which is adapted to receive the valve stem 14. The mounting cup also serves as a closure for the container and carries the sealing gasket in the annular channel 22 which is applied over the opening in the domed top portion 12 of the container.

An axial sectional view of mounting cup 18 is shown in FIG. 2 in inverted position relative to its placement in the assembled container. The cup comprises a circular panel 19 having an integral skirt 20 depending from its periphery. The free edge of skirt 20 outwardly flanged at 21 to form an annular gasket-receiving channel 22. The inner portion of panel 19 is countersunk to form a tubular recess, generally designated at 23, which has a dependent circular wall 24 integrally joined with an apertured horizontal wall 25. When the cup is placed in sealing position, the tubular recess 23 acts as a pedestal for the valve unit and the valve stem is admitted into the container through apertured wall 25. The gasket 26 is disposed predominantly in the annular channel 22 of the cup.

FIG. 3 shows the gasketed mounting cup of FIG. 2 crimped in position over the mouth of an aerosol container. As illustrated in FIG. 3, the open end of domed portion 12 of the container is provided with an outwardly curled peripheral bead 27 which defines the container mouth. The annular channel 22 of the mounting cup embraces the bead 27 of the container so that the gasket 26 carried by channel 22 is positioned on bead 27. The lower portion of the skirt 20 is flared outwardly against the wall of domed

CDD12506



portion 12 adjacent to the bead 27. The gasket forms a seal between the skirt and the wall adjacent to the container mouth. The crimping dimensions as measured from swaged seam 28 are identified on the drawing as the crimp depth and the crimp diameter.

#### DETAILED DESCRIPTION OF THE INVENTION

The compositions that are used in the process of this invention consist essentially of a low molecular weight polyurethane prepolymer into which there is dispersed a finely divided polyamine carbamate. Fillers, solvent, antioxidants, pigments and other additives for elastomers, which are free from active hydrogen, may be incorporated for the conventional effects. The usable prepolymers are low molecular weight reaction products of polyisocyanates, preferably aromatic or alicyclic, with polyfunctional organic compounds containing active hydrogen, preferably polyhydroxy compounds. If desired, the isocyanate groups of the prepolymer themselves may be blocked, for instance with nonylphenol, to be freed later by heat, for reaction with the polyamine and moisture. The best results have been obtained with mixtures of triols and diols in which triol compounds constitute at least 30% of the total polyhydroxy compound mix weight.

The carbamates employed with the prepolymers are, as already mentioned, the reaction products of polyamines and carbon dioxide under anhydrous conditions. The compounds so prepared are generally stable solid materials which may be pulverized for dispersion into the polyurethane prepolymer or dispersed in an organic non-aqueous inert solvent prior to admixture with said prepolymer. The preferred particle size of the carbamate for mechanical stability of the prepolymer mix and the uniformity of the cured polyurethane mass is such that the powder should pass through a 200 mesh or finer U.S. Bureau of Standards screen. Typical examples of usable carbamates are provided in U.S. Pat. Nos. 3,029,227 and 3,344,175.

In the process of the invention, the annular channel of aerosol mounting cups is lined with the prepolymer-carbamate composition by means of appropriate high speed gasket lining machinery. This operation may proceed at rates of 150 or more cups per minute per lining station. After lining, the cups are routed through an oven in which the gasketing material is subjected to a sufficiently high temperature, e.g. 175.degree.F, to decompose the carbamate into its component polyamine and carbon dioxide. In a matter of minutes, usually 5 or less, the polyamine reacts with the prepolymer and gels it. Surprisingly, the carbon dioxide released does not adversely affect the relatively thick channel-confined aerosol gasket to an undesirable degree. The lined cups can be examined at that point for gasket defects and those acceptable are immediately packed for shipment to the aerosol packager. Complete cure of the gaskets subsequently takes place, without further intervention, through reaction of the polymer with ambient moisture during a period of 3 to 5 days.

The temperature at which the gasketing mixture will gel depends to a certain extent upon the nature of the polyhydroxy compound mix as well as on the decomposition point of the carbamate. Thus, although a temperature within the range of 150.degree. to 300.degree.F or higher can be used, gelling can generally be effected in about 2 minutes at temperatures under 200.degree.F.

One of the principal advantages of this method of lining cups over conventional methods lies in the early possibility of examining the cups for satisfactory lining. With a conventional aerosol gasketing system such as one based on polychloroprene for instance, a 5 to 6 hour curing cycle is required involving various stages of drying and curing in a convection type drying and curing oven. Thus, in order to produce gasketed mounting cups at high rates, the drying and curing process must be of exceptionally large capacity and, furthermore, the final production results cannot be fully observed

CDD12507

until tens of thousands of units or more have become involved. In such circumstances defects in composition or mechanical performance may be quite costly. With this in mind, it can readily be understood that the ability to evaluate the quality of the final product after only five minutes is a great improvement over the 5 or 6 hours processing delay normally experienced.

The process of the invention can be further advantageously characterized by a great reduction, if not complete elimination, of the quantity of solvents and other volatile products usually associated with gasketing compositions. A conventional polychloroprene composition, for instance, can contain up to 60% by weight of high boiling aromatic solvent, not to mention various additives such as plasticizers, curing agents and the like which either must be removed during the drying or are partly entrained or decomposed to noxious substances during the extended drying and curing process.

Subject to the preferences already expressed in terms of prepolymers, i.e. partial reaction products of cyclic diisocyanates with mixtures of diols and triols, such as polyalkylene glycols and polyhydroxy polyethers, there may be used other polyisocyanates and polyhydroxy compounds to achieve useful, if not optimum properties in the gaskets.

Among the polyisocyanates that may be employed in this manner are: the various unsubstituted phenylene diisocyanates, as well as those having one or more substituents such as methyl and other lower alkyl groups having up to about 4 carbon atoms, halogen atoms, nitro groups, alkoxy and aryloxy groups; various substituted and unsubstituted biphenylene diisocyanates; substituted and unsubstituted diphenyl diisocyanates such as the diphenylmethanes-, the diphenylisopropylidenes- and the diphenylsulfone diisocyanates; the naphthalene diisocyanates; alkylene diisocyanates containing up to about ten alkylene carbon atoms; cycloalkyl diisocyanates containing one or more cycloalkane rings such as 1,3-cyclopentene- and 4,4'-dicyclohexylmethane diisocyanate; and other diisocyanates such as 1-ethyleneisocyanato-4-phenyl-isocyanate. Examples of tri- and tetra- isocyanates that may be used include the benzene and toluene triisocyanates, 2-methyl-2-(4-isocyanatophenyl)-trimethylene diisocyanate, tetrakis-(4-isocyanatophenyl)methane and bis-(4-methyl-2,5-isocyanatophenyl) methane. Polymeric materials such as polymethylene polyphenylisocyanate may also be employed.

Suitable compounds that will react with the above isocyanates to yield the necessary prepolymers include polyols such as ethylene glycol, glycerol, pentaerythritol, sorbitol, triethanolamine, as well as polymeric compounds such as polyglycols, polyhydroxy polyesters, polyhydroxy polyesteramides and polyhydroxy polyether oils.

Typical polyglycols include polyalkylene glycols, such as polyethylene glycol, polypropylene glycol, and polybutylene glycol.

Representative polyesters are reaction products of dihydric alcohols, such as ethylene glycol, diethylene glycol, tetraethylene glycol, 1,3-propylene glycol, dipropylene glycol, or higher polyhydric alcohols such as glycerol, trimethylol propane, pentaerythritol, mannitol, or mixtures of two or more of the alcohols with a polycarboxylic acid or anhydride such as succinic, adipic, glutaric, maloric, sebacic, azelaic, phthalic, terephthalic, isophthalic, trimellitic and pyromellitic acids and their anhydrides. Mixtures of the acids and anhydrides can be employed.

Illustrative polyhydroxy polyesteramides are the reaction products of a polyhydric alcohol with a dicarboxylic acid, examples of both of which have been mentioned above, and as necessary, diamines or amino-alcohols such as ethylene diamine, hexamethylene diamine, phenylene diamine, benzidine and monoethanolamine.

CDD12508

Examples of polyhydroxy polyethers include linear hydroxyl-containing polymers and copolymers of cyclic ethers such as ethylene oxide, epichlorohydrin, 1,2-propylene oxide, oxacyclobutane, and tetrahydrofuran, or branched polyethers obtained from the condensation of the aforementioned ethers with branched polyhydroxy compounds such as glycerol, 1,1-trimethylolpropane, pentaerythritol, sorbitol and sucrose. Mixtures of linear and branched polyethers or mixtures of polyesters and polyethers can also be employed.

Other polyhydroxy materials, for example, esters of hydroxycarboxylic acids, such as castor oil and glyceryl monoricinoleate, can also be used.

The solid polyurethane polyurea which results from the curing of the sealing compositions of interest may be crosslinked to any degree desired, depending on the number of functional groups present and the ratio of amino groups to isocyanate groups.

Satisfactory crosslinking of the polymers used in this invention is generally achieved with a carbamate to terminal isocyanate group chemical equivalent ratio of, for instance 0.5:1. These proportions may also vary more broadly depending on the particular nature of the components used and the degree of crosslinking required by the application intended for resulting polymer. Thus, ratios of 0.2:1 to 1.3:1 have been successfully employed with various formulations.

The compositions of the invention will generally contain a solvent to facilitate handling of the prepolymer mix by gasket-laying machinery. Usable solvents are organic liquids that do not possess any active hydrogen to react with the isocyanate groups of the polymer and are capable of either evaporating under process conditions or acting as plasticizers when left in the gasket. Examples of suitable liquids for this purpose include: aromatic hydrocarbons, such as benzene, toluene and xylene; acetic esters, such as ethyl and butyl acetates; chlorinated aromatic and aliphatic solvents, such as monochlorobenzene and carbon tetrachloride; ethers, such as butyl ethyl ether, dioxane and hydrofuran; and ethylene glycol monomethyl ether acetate. Solvents having active hydrogen such as the lower alcohols, may be used to prepare the carbamate, but they must be removed from the dispersion before it is mixed with the prepolymer. Relatively small quantities of solvent are needed for the gasketing compositions used in the process of the invention due to the liquid nature of the prepolymer. Thus, less than 10% by weight is generally sufficient. As a result, the pollution problem created during the drying of conventionally lined mounting cups as well as the precautionary equipment required to solve that problem are either minimized or eliminated. The latter advantage is fully realized when the solvent or diluting liquid selected doubles as a plasticizers and thus need not be removed from the composition. Certain high boiling esters, such as dioctyl and diisodecyl phthalate and the like, serve well in this capacity.

As fillers, there may be used, for example, calcium carbonate, calcium silicate, aluminum silicate, silica, chrysolite *asbestos*, carbon black, titanium dioxide, fully calcined clays and talcum powder. The latter material is preferred.

The following examples are provided to further illustrate the invention. All parts and percentages therein are on a weight basis, unless otherwise specified.

#### EXAMPLE 1

A stable prepolymer-carbamate composition is prepared from the following ingredients:

CDD12509

Component	Parts by Weight
Poly(oxypropylene)glycol, mol. weight	5.8
2,000 (POPS)	
Poly(oxypropylene)triol, mol. weight	35.4
4,000 (POPT)	
Tolylene diisocyanate (TDI)	5.7
Talc	39.9
Diethylenetriamine carbamate (DETAC),	5.7
33% solids	
Xylene	6.4
Silicone Oil	1.1
	100.0

The hydroxy compounds, POPG and POPT, and the talc are first mixed in a steam-jacketed glass lined reactor. The mix is heated to 100.degree.C and the reactor evacuated to remove water. After complete removal of the water, the batch is cooled to 50.degree.C and the xylene is charged and mixed. TDI is then introduced while the reactor is brought to 80.degree.C. The reaction is carried on at that temperature until the isocyanate content of the prepolymer mixture reaches 2.9%. The reaction product is then cooled to 30.degree.C and the DETAC dispersion in e.g. xylene, as well as the silicone oil, are mixed in to complete the batch.

## EXAMPLE 2

The liquid compound prepared according to Example 1 is applied at the rate of about 300 mg solid basis per cup to aerosol valve mounting cups by conventional aerosol gasketing equipment. Production rate for this operation is about 200 cups per minute per application nozzle. After the liquid has been applied to the cups, the latter are transferred by automatic belt feed mechanism to a small capacity oven for the gelling cycle. A "baby" size or "half" size Hurricane oven suffices for this operation. The gelling cycle on the moving belt through the oven lasts two minutes at 160.degree.F for the compound of Example 1. The cups are immediately packaged in shipping boxes. Full cure then takes place through ambient atmospheric moisture within 5 days.

## EXAMPLE 3

To measure the effectiveness of sealing and swaging of the aerosol mounting cups on aerosol cans, tests were carried out on a commercial aerosol filling line with a number of different aerosol cup sealing gaskets. The history of loss of volatile contents of the test cans was recorded and the number of cans which failed to seal was noted.

The test was made at various crimping and swaging conditions, i.e. by varying the crimp diameter and the crimp depth (see FIG. 3 of the drawing). In the following table, the results of a test of cups made with the composition of Example 1 are compared to those of cups made with a polychloroprene compound such as that of Example 3 of U.S. Pat. No. 3,389,113 cured in the conventional manner.

CDD12510

Crimping Condition		
Gasket Compound		
	Carbamate	
	Gelled/Ambient	
Polychloroprene		
	Cure Polyurethane	

I.		
Crimp Depth --		
0.160 inches	0%	0%
Crimp Diameter		
1.054 inches		
II.		
Crimp Depth --		
0.205 inches	14%	2%
Crimp Diameter --		
1.068 inches		

Crimping condition I represents the optimum target conditions presently required to yield 100 percent satisfactory sealing. In this instance, both gaskets performed well. However, maintaining these optimum crimping conditions during high speed filling and sealing of aerosol cans is not always practical and thus some deviation always occurs yielding some cans which are not as tightly sealed as in condition I. These cans belong to crimping condition II. At this condition, the conventional gasket fails to seal more frequently than the carbamate gelled/ambient cure polyurethane aerosol cup gasket. This reduction in failing cans from 14% to 2% represents a significant improvement in sealing effectiveness and reduces the present rate for discarded and scrapped aerosol cans which leak through the crimped seam between the valve mounting cup and can.

#### EXAMPLES 4 TO 7

Other prepolymer-carbamate compositions were prepared for testing as in Example 3. These compositions generally comprised: about 40% of polyhydroxy compounds in various diol to polyol mix ratios; about 6 to 8% polyisocyanate, in these instances--dicyclohexylmethane diisocyanate; about 38% talc; about 5.5 to 6.0% carbamate (DETAC); about 4.5% xylene; and about 1.2% silicone oil. Some of the polyhydroxy compound mixes selected were as follows:

Polyhydroxy Compound Mixtures				
Polyhydroxy Compound				
	Example			
	4	5	6	7
Poly(oxypropylene)glycol, 2000*	3.59	3.76		
			22.85	
				5.26
Poly(oxypropylene)glycol terminated with ethylene oxide, 4000		15.63		21.87
Poly(oxytetramethylene)glycol, 3000	1.76			
Poly(oxypropylene)triol, 3000	9.57	24.53		
Poly(oxypropylene)triol, 4000			4.94	

CDD12511

16.20

Poly(oxypropylene)triol terminated  
with ethylene oxide, 6000

	29.06	14.99
Total Weight Percent	43.98	
	43.92	
	42.78	
	43.33	

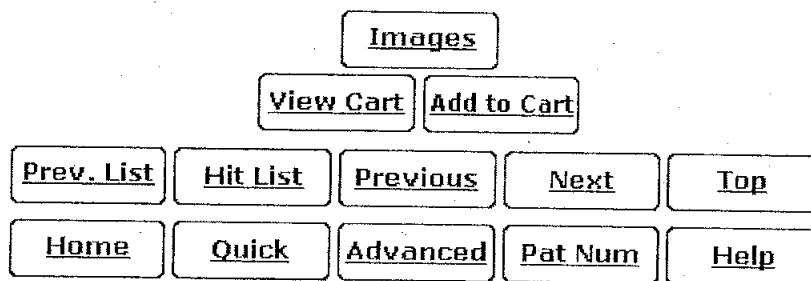
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\*molecular weight

After application to mounting cups and curing, the cups were swaged onto filled aerosol cans under the two crimping conditions of Example 3. Leakage tests again showed a significant improvement in sealing as compared to the conventional polychloroprene system.

In summary, the present invention relates to an aerosol gasketing system which not only benefits from the processing ease attending stable one-package fast-curing polyurethanes, but also performs in a superior manner under the type of conditions generally availing in that field of packaging. Although a limited number of examples have been provided here to illustrate various operational features of the invention, it is apparent that the man skilled in the art can devise several other embodiments which will be substantially within the scope of the invention as described in the appended claims.

\* \* \* \* \*



CDD12512

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( 53 of 59 )

**United States Patent**  
**Shotbolt**

**4,081,503**  
**March 28, 1978**

Method of forming closure liners**Abstract**

Container closures are given the customary sealing gasket of plastics material by a two stage deformation of a pellet of the plastics material. In a first stage the material is partly deformed and is heated by direct conduction from a heated deforming member, and in the second stage a relatively cool final moulding die effects further deformation to impart the desired finished configuration to the gasket. The pressure member, and possibly also a workpiece support on which the upturned closure rests, is heated by means of a radiant heat source directing radiant heat onto a blackened roughened surface of the pressure member, and the workpiece support where applicable. The pressure member has a low-adhesion surface of polytetrafluoroethylene.

**Inventors:** Shotbolt; Keith (Gerrards Cross, EN)

**Assignee:** W. R. Grace & Co. (Cambridge, MA)

**Appl. No.:** 491749

**Filed:** July 25, 1974

**Current U.S. Class:**

264/268; 264/294; 264/296

**Intern'l Class:**

B29D 031/00

**Field of Search:**

264/268,294,296,25

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<u>3509252</u>	Apr., 1970	Baehr	264/296.

CDD12513

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### *Claims*

I claim:

1. In a method of moulding a thermoplastic material into a gasket for a container closure comprising the steps of heating a quantity of the material and deforming said material into a desired finished configuration, the improvement wherein the material in the form of a pellet is placed into the closure and is subjected to two consecutive deformation operations, the first operation being carried out with a heated pressure member which both heats the thermoplastic material and deforms it into a partially moulded configuration, and the second operation being carried out using a positively cooled moulding member, shaped to impart the desired finished configuration to the thermoplastic material, which is maintained at a temperature lower than that of the heated and partially deformed thermoplastic material.
2. The improvement of claim 1 wherein the finished article is a sealing gasket in a bottle closure and the said moulding member has a configuration which imparts to the gasket a cross-sectional form which conforms sealingly with the container to be closed.
3. The improvement of claim 2, wherein the bottle closure rests on a heated support during the first deformation operation.

### *Description*

#### BACKGROUND OF THE INVENTION

The present invention relates to a method for moulding thermoplastic compositions. In a preferred form, the invention relates to the moulding of liners or gaskets in container closures.

Hitherto it has been known for example when forming a liner for a container closure to pre-heat the thermoplastic material and to mould the heated and thus softened material using a mould die which is not itself heated and may even be positively cooled so that in either case it presents a relatively cool moulding surface. However, the above described moulding process involves a considerable dwell time of the thermoplastic material in an oven, through which closures carrying metered quantities of the plastics material, are passed along a tortuous conveyor path. Such a system is, for example, disclosed in our British patent application No. 5948/73 in which the heat is imparted to the thermoplastic material by radiation within the oven. This has the disadvantage that, if for any reason the movement of the closure-carrying conveyor through the oven stops, the closures in the oven rapidly become overheated and "scorched" with the result that they are unsuitable for use.

#### SUMMARY OF THE INVENTION

CDD12514



The present invention proposes to mould thermoplastic material by firstly applying heat directly to the material, which preferably is in the form of a pellet or spot, using a pressure member which exerts pressure on the material to impart thereto an initial deformation, and subsequently moulding the material to final desired dimensions using a die the temperature of which is lower than that of the heated thermoplastic material. The die is thus desirably not heated at all or is positively cooled.

In order to ensure that the thermoplastic material does not stick to the heating member that surface of the pressure member which in use contacts the thermoplastic material may be coated with a medium imparting low-adhesion characteristics. Preferably the coating may comprise polytetrafluoroethylene (P.T.F.E.). More preferably, the particular P.T.F.E. coating employed may be in accordance with the "ARMOURCOTE" system in which the surface of the pressure member is first of all given a sintered stainless steel layer, onto which the subsequently applied P.T.F.E. can more securely be keyed.

By exerting pressure on the pellet or spot while heating it, the thickness of the thermoplastic material is reduced and the area is increased. Thus the rate of transfer of heat is increased markedly. For example, if the diameter of a pellet is doubled the heat transfer is increased by 16 times.

The invention also proposes apparatus for applying a moulded surface covering of thermoplastic material to a work-piece, comprising a work-piece support for supporting the work-piece, means for feeding thermoplastic material into contact with the work-piece, a pressure member mounted and drivable for movement in a direction towards a supported work-piece so that the pressure member bears firmly against and softens and thins the thermoplastic material, means for imparting heat to the pressure member and a moulding die member adapted to bear against the softened thermoplastic material and impart the desired final dimensions thereto.

Preferably, the pressure member is given a low-adhesion surface such as P.T.F.E.

Desirably the apparatus may include a rotary turret incorporating a plurality of workpiece supports and a plurality of separate pressure members, all associated with a common radiant heat source and arranged on a circular arcuate path around the axis of rotation of the turret. The workpiece can be supported on a respective one of the workpiece supports and the associated pressure members will be driven down into contact with thermoplastic material on the workpiece to impart heat thereto while causing initial deformation of the thermoplastic material.

Advantageously, means may be provided for heating the workpiece supports, so that the workpieces themselves are heated thereby.

Conveniently the means for heating the pressure member and/or the workpiece support may comprise a source of radiant heat located adjacent the pressure member. Advantageously, the pressure member includes a blackened surface disposed directly opposite the radiant heat source which, in a preferred form, may be an infra-red electrical heating element which can be caused to glow when energised.

Other alternative forms of heating the pressure member and/or the workpiece supports may be employed. For example the heat may be imparted by direct conduction from electrical cartridge heaters, or by playing a gas flame on the heating member and/or the support. A stripper member may be located above the workpiece path to hold down the workpiece as the pressure member is withdrawn after hot pressing the plastics material. Where the workpieces are container closures the stripper member is a plate with a slot extending peripherally of the turret to receive the pressure member, in this case a plunger which may have flats formed to enter the slot while the hot pressing

CDD12515

operation is carried out by a moulding head below the stripper plate which may or may not have similar flats, as desired.

The heating turret may include separate temperature sensors for detecting the temperatures of the workpiece supports and the temperatures of the pressure members and a separate control system may desirably be provided for controlling the temperatures of the supports and the pressure members to maintain optimum heat transfer conditions.

This heating turret may be fed from an insertion device, such as a further turret of the form disclosed in our copending application No. 5948/73, and the heating turret may be arranged to deliver the workpiece with partly deformed plastics material directly to a further moulding turret.

The workpieces may suitably be preheated before insertion of the plastics material by means of a rotary preheating turret in which the workpieces rest on hot supports. The hot supports may be heated by electrical cartridge heaters, gas flames or the like. Advantageously the supports may be heated by means of conduction from a hot receptor arranged to receive heat from a stationary radiant source such as an infra-red heating element.

By avoiding the conventional convective or direct radiation heating of the plastics material, it is possible to impart to the plastics material the desired softness together with an advantageous pre-shaping in a time which, when moulding liners or gaskets in container closures, can be reduced from the order of 25-40 seconds down to the order of 2.5 seconds. In the event of shut down of the machine while the heating turret is loaded, very few if any closures will be rendered unusable, because they are not subjected to intense heat.

Similarly the use of direct conduction for preheating the empty workpieces means that heat can be transferred quickly into the workpiece from a large capacity heat-transmitting conductor member without the need for fierce radiation directly onto the workpiece surface since this radiation tends to mar some lacquers used on container closures.

The main heating turret used in the preferred embodiment of apparatus according to the invention is far more compact than the previously used ovens.

#### BRIEF DESCRIPTION OF THE DRAWINGS

In order that the present invention may more readily be understood the following description is given, merely by way of example, reference being made to the accompanying drawings in which:

FIG. 1 is a schematic top plan view of apparatus for lining container closures in accordance with the present invention;

FIG. 2 is a vertical sectional view through one of the positions on the heating/pressing turret;

FIG. 3 is a horizontal section on the line III--III of FIG. 2; and

FIG. 4 is a view similar to FIG. 2 but showing an alternative form of heating/pressing turret;

FIG. 5 is a plan view of the stripping plate shown in FIG. 4;

CDD12516

FIG. 6a and 6b are a side elevational, partly sectional and an end elevational view, respectively, of the plunger bottom pad shown in FIG. 4.

#### DESCRIPTION OF PREFERRED EMBODIMENTS

Referring now to FIG. 1, the closures to be lined are advanced along a schematically illustrated chute conveyor generally designated 1, to a feed star wheel 2 from which the stream of empty closures is fed to a preheating turret 3 to be preheated by direct conduction of heat from a closure support. The closures passing around the turret 3 are heated to a temperature sufficient to cause subsequently inserted pellets of thermoplastic material to adhere securely to the inner face of the closure for subsequent operations.

From the preheater turret 3 the closures pass around a first transfer star wheel 4 and are fed to a pellet insertion turret 5, preferably of the form disclosed in our copending British patent application No. 5948/73.

The star wheel 4 has a plurality of pockets which advance the closures while the disc-like end walls of the closures ride on a smooth guide rail which has a small contact area with the closures so as to minimize heat loss from the already preheated closures and to avoid scratching the decorative lacquer which will already have been applied to the outer surface of the closure.

Individual pellets of thermoplastic material are introduced to respective ones of the preheated closures by turret 5 before the closures are transferred by means of a similar secondary transfer star wheel 6 onto a turret 7, where they are to be heated and initially deformed, as will be described in more detail below.

From the heating turret 7 the closure, which now have soft flattened plastics disc therein, are transferred, via a third similar type of transfer star wheel 8, to a moulding turret 9 where mould dies considerably cooler than the thermoplastic material impart the final desired shape to the thermoplastic material, before the closures then advance to an inspection station along the same or a different conveyor as shown at 10.

In FIG. 2, one station 11 on the heater turret 7 is shown as comprising a vertically movable pressure member in the form of a heating plunger 12 having an upper part 12a of an *asbestos* impregnated phenolic resin known by the Trade Name Ferobestos. At the top end of the upper part 12a is the cam follower roller 25 mounted in a bifurcated carrier 26 which is spring urged upwardly by a helical compression spring 27 into contact with a circumferential cam 16.

The heating plunger 12 carries with it a heat-absorption member 13 having an arcuate surface 14 arranged on a circular arc about the centre of a thermostatically controlled heating element 15 which itself extends around the circumference of the main turret.

The circumferential cam 16 at the top of the machine provides the means for causing periodic raising and lowering of each plunger 12 relative to a container closure resting on a support member 17, so that a removable bottom pad 18 of the plunger enters the closure to compress the pellet of a thermoplastic material, in this case a composition consisting of polyethylene and butyl rubber as disclosed in British Pat. Nos. 1112023, 1112024 and 1112025. Amongst many other suitable materials is polyvinyl chloride. Also suitable are the mixtures of polyethylene and styrene butadiene as disclosed in British Pat. Nos. 1196125 and 1196127.

CDD12517

This action imparts heat to the pellet allowing it to soften and at the same time to spread due to the downward compression of the plunger. By the end of the heating and compression operation the entire body of the plastics material is soft and ready for a subsequent moulding operation using a relatively cold mould at the moulding turret 9.

The arcuate surface 14 of the heat receiving member 13 is both blackened and roughened so as to improve its heat-absorption characteristics with respect to radiant heat, and a reflector 19, extending around the circumference of the turret, throws back radiant heat against the surface 14. The heat picked up by the surface 14 is then conducted through the heat absorption member 13 to the plunger 12 so as to maintain the bottom surface 18 at a temperature of approximately 200.degree. C to impart the necessary heat to the plastics material.

The closure-supporting member 17 is provided with a similar heat-absorbing member 20 mounted close to a second separately thermostatically controlled heating element 21 and a second reflector 22 so that the underside of the closure is also heated to assist in transferring heat to the pellet.

The entire turret is driven for rotation in synchronism with the remainder of the apparatus.

As shown in the top plan view of FIG. 3, the various heat absorbing members for the closure support members and the plungers are angularly spaced from one another to avoid any problems of circumferential expansion in the range of temperature encountered between room temperature and the operating temperature of around 200.degree. C.

Compressing the pellet as it is heated ensures that there is improved contact area between the plastics material and the heated surface of the plunger and the surface 18 is coated with the "Armourcote" technique using sintered stainless steel as a keying surface for P.T.F.E. The portion of the plunger 12 carrying the P.T.F.E. may be detachable, so that it can be replaced should the P.T.F.E. become worn.

The heating system of the preheater turret 3 is similar to that of the main heating/pressing turret 7 in that the closure supports are heated from below by radiation from a thermostatically controlled heating element with a reflector to throw back the radiant heat towards a blackened heat-receiving surface of the closure support.

A stripping plate (not shown) is provided just above the workpiece path around the turret 7 at the position where the plungers are withdrawn from the closures, this stripping plate having an arcuate slot through which the plunger extends slidably. As the plungers are withdrawn upwardly the rims of the closure skirts will strike the underside of the stripper plate to hold the closures down to effect separation of the plungers from the heated plastics material.

FIG. 4 shows a vertical sectional view similar to that of FIG. 2, but depicts a modified form of the heating plunger construction for use on the main heating/pressing turret 7.

Since many of the components of the turret of FIG. 4 are identical to those of FIG. 2, the same reference numerals have, in many cases been employed. Where the reference numerals in FIG. 4 have been primed, they denote modified elements which may be analogous to the corresponding elements of FIG. 2.

The plunger 12' in this embodiment of the turret co-operates with a slotted stripping plate 28 which

CDD12518

extends horizontally above the path of the closure support members 20 at the part of the heating/pressing turret where the plunger 12' is to rise away from the partially moulded plastics composition in the container closure 30.

The stripping plate 28 acts in the same manner as the non-illustrated stripper plate of FIG. 2 to hold down the closure 30 since the rim 31 of the skirt of the closure will rise to strike the underside of the stripping plate 28 as the plunger is withdrawn and further withdrawal of the plunger 12' will raise the bottom pad 18' away from the heated and partially formed plastics composition in the bottom of the closure since the adhesion of the plastics composition to the bottom of the closure is stronger than its adhesion to the bottom pad 18', by virtue of the coating of polytetrafluoroethylene on the underside of the pad 18'.

In order to allow this co-operation between the stripper plate 28 and the plunger 12', the plunger and the bottom pad both have flats 29 formed in their lateral surfaces and these flats leave the plunger 12', 18' with a transverse dimension which is considerably less than the diameter parallel to the flats. These flats allow the plunger to be withdrawn upwardly into the slot of the stripper plate so as to release the closure for radially outward removal from the press/heating turret 7 at the third transfer star wheel 8 for advancing to the moulding turret 9.

After this stripping action has been completed, the plunger 12' will continue its travel around the turret 7 and will pass out of the end of the slot 43 in the stripper plate 28 after the closure 30 has been separated from the heating pad 18'.

This flattened circular form of pad 18' is particularly advantageous where, as illustrated in FIG. 4, the closures 30 are of the deep drawn type without a beaded rim to the skirt, since the transverse dimension of the stripper plate slot will be as small as possible so that the delicate closure rim bears against the stripper plate over as great as possible a part of its periphery thereby distributing the end loading on the skirt rim more evenly.

As illustrated in FIGs. 6a and 6b, the mounting system for the bottom pad 18' involves a stud 32 of unthreaded form on the pad, but having at some point along its length a peripheral groove 33 whose lower wall is defined by a radially extending planar face and whose upper wall is of upwardly divergent conical form when in situ. A grub screw 34 extends radially inwardly of the plunger 12', as shown in FIG. 4, and a similar screw extends inwardly from a diametrically opposite position so that these two screws both abut the upwardly divergent conical wall of the groove to pull the stem 32 upwardly as the two grub screws 34 are tightened. In this way, a secure clamping action can be exerted on the bottom plate 18' independently of the particular orientation of the pad 18' about the vertical axis.

Although not shown in detail in FIG. 2, the bottom pad 18 is circular and has a threaded stem which is screwed into a threaded socket in the bottom end of the plunger 12. Such an arrangement requires a widening at the downstream end of the stripper plate slot to accommodate the circular plunger. The initial separating action of the plunger and closure occurs at a narrower upstream portion of the slot where flats in the plunger body allow the plunger body to pass along the slot even though the bottom pad 18 is wider than this part of the slot and is still disposed in the closure below the narrow slot portion. The arrangement in FIG. 4 is preferably where flattened bottom pads 18' are to be used.

The side elevational, partly sectional, view of FIG. 6a illustrates the profile of one form of pad where the end face is slightly concavely dished.

Temperature control for the heating elements 15 and 21 is effected by means of a thermocouple 35 which picks up radiant heat from the heat-collecting member 13 for the plunger 12', and a further thermocouple 36 for sensing the temperature of the closure support bosses 17. The thermocouple 35 is effective to control the power supply to heating element 15 for maintaining accurate temperature stability of the plunger 12' and the thermocouple 36 is effective to control the power supply to the lower heating element 21 for ensuring temperature stability of the closure support 17.

During rotation of the turret all the various closure support members 17 and all the various heating members 13 will pass the temperature monitoring station so that the power supply to the heaters will vary in response to the mean temperature, around the turret, of these respective sets of elements.

A further difference between the turret of FIG. 2 and that of FIG. 4 is that the peripheral cam no longer acts purely by virtue of its downwardly facing edge surface against which the roller 25 is spring urged, but instead in FIG. 4 the cam 16' has a cam slot formed in one edge to receive the cam follower roller 25' which in FIG. 4 is mounted on the side of the plunger body at the top end of the plunger. This dispenses with the need for a spring return action since the slot can now both drive the plunger downwardly and lift the plunger up again thanks to the side mounted cam follower roller configuration.

Furthermore, whereas in FIG. 2 the upper plunger portion 12a was formed of the *asbestos*-impregnated phenolic resin material known by the trade name Ferobestos, chosen for its temperature stability characteristics, it has been found convenient to adopt a different arrangement in the embodiment of FIG. 4. Here the plunger 12 has a stainless steel main body portion 37 with an integral threaded upper shaft portion 38 at the upper end of this portion 37. The threaded shaft extends as a clearance fit through the carrier 39 on which the cam follower roller 25' is secured in a side-mounted configuration.

The heat-receiving member 13 is clamped to the bottom end of the stainless steel body portion 37 by means of a cap screw 44 which bears against a heat-insulating bush 45 of the thermal insulating material known by the trade name Sindanyo. This bush 45 is received in a bore 46 of the heat-receiving member 13, and a further heat insulating bush 47 is placed between the stainless steel main body portion 37 of the plunger and the upper side of the heat-receiving member 13. Total thermal insulation of the plunger body 37 from the heat-receiving member is completed by an air gap 48 between the screw 44 and the heat-receiving member.

Above and below the carrier 39 are nuts 40 and 41 which can be adjusted in their position up and down the threaded shaft 38 so as to set precisely the vertical distance between the upper face of the cam groove in cam 16' and the lower extremity of the bottom plunger pad 18' at the lobe of the cam. This is clearly an important dimension in the machine since even the smallest inaccuracy in the spacing between the top face of the closure support 17 and the flat underneath face of the pad 18' will provide a considerable variation in the degree of pressing to which the plastics material is subjected during the heating operation. The extent of this squashing action is particularly important in view of the fact that the heat exchange contact area between the bottom pad 18' and the plastics material varies as the square of the radius of the squashed plastics mass. The carrier 39 may be formed of any suitable material. For example it may be formed of Ferobestos or may be injection moulded from a plastics material.

Thus, once the turret has been assembled it is rotated step by step between successive adjustment

CDD12520

stages in which, as each plunger 12' is brought into line with the lobe of the cam 16', the clearance between the flat bottom face of the pad 18' and the flat upper face of the closure support 17 is checked and if necessary adjusted by moving the shaft 38 upwardly or downwardly relative to the carrier 39 by virtue of the adjusting nuts 40 and 41.

Apart from the cam operated fully automatic turrets illustrated in FIGS. 2 and 4, it has also proved advantageous to construct a laboratory test rig in which a pellet of plastics material is placed manually in an upturned closure using a hand-held insertion tool operated in a similar manner to the inserter disclosed in our copending application No. 5948/73, and for this pellet of plastics material to be moved into register with a press/heating plunger similar to the plunger 12 of FIG. 2, or the plunger 12' of FIG. 4, and then for the closure with its partially pressed plastics composition to be moved to a moulding station where a relatively cold final moulding tool is brought down manually with the assistance of lever action to compress the plastics material into its final moulded configuration. Suitable means for pre-heating the closure before insertion of the plastics material will, of course, be employed even if not incorporated in the actual laboratory rig.

In this way it is possible for the various parameters of the moulding process, for example the composition of the plastics material, the pre-heating temperature, the configuration or temperature of the press/heating mould, the configuration of the moulding face of the cold mould tool and many other variables to be changed relatively easily so as to ascertain the optimum conditions for any particular type of closure. In this way the development work can be carried out on a single station machine and the lessons from this development work can subsequently be incorporated in a multistation machine similar to the turrets illustrated in FIG. 2 and FIG. 4.

The advantages of the direct conduction form of heating for the plastics composition as proposed by the present invention include the fact that the heat is now applied directly to the top face of the plastics composition by conduction, and the rate of supply of heat increases during the pressing operation because of the progressive increase in the contact area between the squashed plastics composition and the bottom pad 18, or 18' of the plunger 12 or 12'.

This improvement of deforming while heating the plastics composition assists in ensuring a more easily mouldable state for the plastics material which, on arrival at the final cold moulding station, has approximately the desired finished diameter. The cold mould now only has to carry out the final shaping operation so a much thinner centre panel can be provided in the finished gasket.

Furthermore, with the press/heating step of the present invention there is no incidence of a "shadow" of the top surface of the initial pellet in the finished gasket. This phenomenon was apparently due to the fact that the cold mould initially contacts the top face of the soft but undeformed pellet to "freeze" it so that during a subsequent moulding operation the material which was initially at the top of the pellet is never adequately deformed. With the present invention the cross-section of the pellet grows during heating and thus when the cold moulding tool impinges on the pre-pressed pellet or disc the whole of the top surface of the disc starts at a uniform temperature and finishes up at a uniform temperature immediately after moulding.

Furthermore, whereas in the past the final cold moulding tool configuration was contoured to give the finished gasket a central portion contoured in the form of a plurality of concentric circular ribs and grooves in order to disguise the unpleasant appearance of the "shadow," the fact that this shadow no longer appears makes it possible for the gasket thickness to be considerably reduced in that these ribs and grooves may be eliminated if desired. This in turn provides greater economy of plastics material,

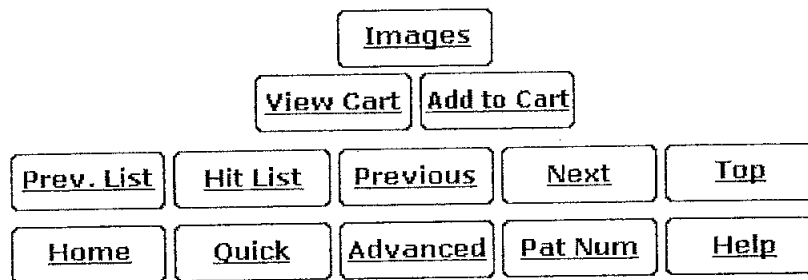
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an important saving having regard to the spiralling cost of plastics materials generally. However grooves or ribs may be included to give the centre panel of the gasket a more pleasing shape if desired.

The above described process is particularly suitable for use with bottle caps, such as crown caps or other caps and is especially suitable for deep drawn caps or the very deep drawn caps known by the trade name Stel Caps.

It will be appreciated that the above detailed description is intended merely to illustrate the operation of preferred forms of the invention and that several modifications can be made to the details set forth without departing from the spirit of the invention.

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<a href="#">Images</a>				

( 47 of 59 )

United States Patent  
Bleyle

4,119,746  
October 10, 1978

Cross-linking resin saturant and method

Abstract

Porous, fibrous materials such as non-woven polyester or polypropylene web materials, cotton web, paper and other materials are saturated with a storage stable one-package latex composition. The latex composition is modified with a temperature sensitive cross-linking agent and neutralizing agents. The latex composition is tailored such that a fibrous material may be impregnated and dried at controlled temperature whereby a thermoplastic resin saturant web intermediate stock material having thermoset properties is produced. The cross-linking system in the latex composition is later activated in a specific manufacturing process at heat forming and molding temperatures to obtain a specifically shaped manufacture of desired rigidity and flexibility.

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**Field of Search:** 260/29.4 UA,29.6 TA 428/290 427/381,385 B,389,391,392  
264/137

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### *Claims*

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What I claim is:

1. The method of preparing a resin impregnated non-woven web having thermoplastic characteristics at temperatures below 190.degree. F. and thermoset characteristics at temperatures above 190.degree. F. comprising:

(a) preparing a cross-linking latex composition by

(1) charging a reaction vessel with a small amount of a seed latex polymer along with an initiator in an aqueous phase to provide nucleating sites for polymerization,

(2) adding, substantially continuously, monomers and emulsifiers at a controlled rate, said monomer consisting essentially of about 59 parts by weight styrene, about 41 parts by weight ethylacrylate, and 2 parts by weight N-methylolacrylamide, the addition taking place at about 150.degree. F. over a 41/2 hour period,

(3) post cooking the latex composition up to about three additional hours to reduce the free monomers content of the latex composition,

(4) adjusting the latex composition after polymerization is completed from as manufactured state to a pH value between 6.0 and 8.0 with a neutralizing agent;

(b) saturating a non-woven web with the adjusted latex composition;

(c) drying the saturated web at a temperature below about 190.degree. F. whereby an impregnated thermoplastic non-woven web having thermoset properties above 190.degree. F. is produced.

2. The method of claim 1 wherein the neutralizing agent is selected from ammonium hydroxide,

sodium hydroxide, potassium hydroxide, morpholine, methylamine, dimethylamine and mixtures thereof.

3. A method of preparing a resin impregnated non-woven fibrous web having thermoplastic characteristics at temperatures below 190.degree. F. and thermoset characteristics at temperatures above about 190.degree. F. comprising:

(a) providing a storage stable one-package polymerized latex composition consisting essentially of the polymerization reaction product of

(1) from about 40 to about 70 parts by weight of a first monomer selected from styrene, vinylidene chloride, methylmethacrylate, butylmethacrylate and mixtures thereof,

(2) from about 30 to about 60 parts by weight of a second monomer selected from methylacrylate, ethylacrylate and butylacrylate, propylacrylate, 2-ethylhexylacrylate, mixtures thereof, and,

(3) from about 0.5 to about 6.0 parts by weight of N-methylolacrylamide;

said composition having been treated with a neutralizing agent after completion of polymerization reaction to prevent cross-linking below temperatures of 190.degree. F.;

(b) applying said neutralized composition to a non-woven fibrous web to impregnate the web, and

(c) drying the impregnated non-woven fibrous web at a temperature below 190.degree. F.

4. The method of claim 3 wherein the neutralizing agent is selected from ammonium hydroxide, sodium hydroxide, potassium hydroxide, morpholine, methylamine, dimethylamine and mixtures thereof.

5. The process of claim 4 wherein the first monomer is 59 parts by weight and is styrene, the second monomer is 41 parts by weight and is ethylacrylate, and N-methylolacrylamide is 2 parts by weight.

6. A process for forming a thermoplastic heat stable stock material for later cross-linking by saturating a non-woven web with a cross-linking resin saturant having cross-linking reaction activation temperature above 190.degree. F. comprising the steps of:

(a) preparing a polymerized resin composition consisting essentially the polymerization reaction product of:

(1) from about 40 to about 70% by weight of a first monomer selected from styrene, vinylidene chloride, methylmethacrylate, butylmethacrylate and mixtures thereof,

(2) from about 30 to about 60% by weight of a second monomer selected from methylacrylate, ethylacrylate, butylacrylate, propylacrylate, 2-ethylhexylacrylate, and mixtures thereof, and,

(3) from about 0.5 to about 6.0% by weight of N-methylolacrylamide;

said composition having been treated with a neutralizing agent upon completion of polymerization reaction to prevent cross-linking below 190.degree. F.;

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- (b) saturating a non-woven web with the resin from step (a);
- (c) drying the resin saturated web at a temperature below the cross-linking temperature of the resin whereby a thermoplastic stock material having thermoset properties is produced.
7. The process of claim 6 wherein the first monomer is 59% and is styrene, the second monomer is 41% and is ethylacrylate, and N-methylolacrylamide is 2%.
8. The process of claim 7 wherein the neutralizing agent is selected from ammonium hydroxide, morpholine, potassium hydroxide, sodium hydroxide, methylamine, dimethylamine and mixtures thereof.
9. A process for treating a non-woven web which comprises:
- (a) contacting said web with a thermosetting latex saturant composition having thermoplastic characteristics at temperatures below about 190.degree. F. when dried on a non-woven fibrous material consisting essentially of a polymerization reaction product of:
- (1) 40 to 70 parts by weight of a first monomer selected from styrene, vinylidene chloride, methylmethacrylate, butylmethacrylate and mixtures thereof,
  - (2) 30 to 60 parts by weight of a second monomer selected from methylacrylate, ethylacrylate, butylacrylate, propylacrylate, 2-ethylhexylacrylate and mixtures thereof, and
  - (3) 0.5 to 6 parts by weight of N-methylolacrylamide, said product buffered to a pH range of from about 6.0 to about 8.0 with a neutralizing agent after completion of polymerization reaction;
- to impregnate said web with said composition, and
- (b) drying the impregnated web at temperatures below about 190.degree. F. whereby the impregnated web has thermoplastic characteristics.
10. The process of claim 9 including the additional step of thermally cross-linking the impregnated web by heating to a temperature above 190.degree. F. to thermoset said composition on said web.

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### *Description*

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#### FIELD OF INVENTION

This invention relates to latex cross-linking web saturants particular suited for impregnating porous, fibrous materials to improve the physical properties thereof and to the impregnated products produced thereby. In a particular aspect, the invention concerns a novel impregnating composition which is comprised of a latex composition modified with N-methylolacrylamide. The modified latex is buffered from a manufactured acid state to a neutral state with a neutralizing agent to prevent premature cross-linking in storage and during the drying of the saturated webs.